भारतीय मानक
Indian Standard

IS 516 (Part 11) : 2020

दृढ़ीकृत कंक्रीट — परीक्षण पद्धतियां

भाग 11 दृढ़ीकृत हाईड्रॉलिक सीमेंट कंक्रीट में पोर्टलैंड सीमेंट की मात्रा ज्ञात करना

(*पहला पुनरीक्षण*)

Hardened Concrete — Methods of Test

Part 11 Determination of Portland Cement Content of Hardened Hydraulic Cement Concrete

(First Revision)

ICS 91.100.30

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Cement and Concrete Sectional Committee, CED 02

FOREWORD

This Indian Standard (Part 11) (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Cement and Concrete Sectional Committee had been approved by the Civil Engineering Division Council.

Testing plays an important role in controlling the quality of cement concrete work. Systematic testing of the raw materials, the fresh concrete and the hardened concrete, is an inseparable part of any quality control programme for concrete. This helps achieve a higher efficiency of the materials used and greater assurance of the performance of the concrete, in regard to workability, strength and durability. The test methods used should be simple, direct and convenient to apply. This standard was formulated with this objective in view.

This standard was first published in 1959. In this revision, it was decided to review and update the various existing test methods of concrete taking into consideration the latest international practices and developments in this field in the country, and also introduce certain new test methods wherever required. In the process, the various existing test methods covered in IS 516 : 1959 'Method of tests for strength of concrete' have been revised. The revision of this standard has been brought out taking into consideration primarily the corresponding ISO standards while also examining the other best practices world over and in the country. In addition, test methods for determination of additional properties have been included in areas such as permeability, initial surface absorption, corrosion of reinforcement, carbonation of concrete (field test), accelerated carbonation test, and, creep of concrete. Also, for better understanding and implementation, some of the other test methods which were spread over in number of other Indian standards have been brought together under the fold of IS 516 as its various parts, such as the splitting tensile strength, ultrasonic pulse velocity test, rebound hammer test, bond in reinforced concrete, and determination of water soluble and acid soluble chlorides. This is with a view to making the standard complete in all respects, and rendering it a comprehensive source of provisions for testing of concrete and reference in other Indian Standards.

In this revision, IS 516 is being split into twelve parts. The other parts in this series are:

Part 1 Determination of strength of hardened concrete
Part 2 Determination of properties of hardened concrete other than strength
Part 3 Making, curing and determining compressive strength of accelerated cured concrete test specimens
Part 4 Sampling, preparing and testing of concrete cores
Part 5 Non-destructive testing of hardened concrete
Part 6 Determination of drying shrinkage and moisture movement of concrete samples
Part 7 Determination of creep of concrete cylinders in compression
Part 8 Determination of modulus of elasticity in compression
Part 9 Determination of wear resistance
Part 10 Determination of bond in reinforced concrete
Part 12 Determination of water soluble and acid soluble chlorides in hardened mortar and concrete

This standard (Part 11) covers two procedures for determination of Portland cement content of a sample of hardened hydraulic cement concrete made with ordinary Portland cement, through determination of soluble silica and through determination of calcium oxide.

This test method shall be applicable as and when published, in place of the corresponding provisions given in IS 1199 : 1959 'Method of sampling and analysis of concrete'. IS 516 : 1959 shall be superseded after the publication of all the parts of the standard.

This revision has been brought out to incorporate the modifications found necessary in the light of experience gained in its use and also to bring it in line with the latest development on the subject. Significant modifications in this revision include,

a) petrographic examination of the concrete sample is to be conducted before hand, to assess the possible interferences and to ensure logical interpretation of results; and

(*Continued on third cover*)

Indian Standard

HARDENED CONCRETE — METHODS OF TEST

PART 11 DETERMINATION OF PORTLAND CEMENT CONTENT OF HARDENED HYDRAULIC CEMENT CONCRETE

(First Revision)

1 SCOPE

1.1 This standard (Part 11) specifies the procedures for determination of Portland cement content of a sample of hardened hydraulic cement concrete. The test method is applicable only for concrete made with ordinary Portland cement.

1.2 The test method consists of following two procedures:

a) Determination of soluble silica, and
b) Determination of calcium oxide.

NOTES

1 Each procedure requires a substantial degree of chemical skill. Determined cement contents are equal to or slightly greater than actual values except for the influence of known interferences.

2 This method has limited applicability when applied to concrete made with blended cements or mineral admixtures. The method gives better results when the samples of cement and aggregates used in concrete are available.

2 REFERENCE

The standard listed below contain provisions, which through reference in this text, constitute provisions of this standard. At the time of publication, the edition indicated was valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent edition of the standard indicated below:

IS No.	*Title*
4032 : 1985	Method of chemical analysis of hydraulic cement (*first revision*)

3 INTERFERENCES

Many constituents and factors of concrete may interfere with the analysis of concrete for Portland cement content. For example, rock minerals or mineral admixtures interfere with the Portland cement content determination to the extent of their solubility during dissolution procedures used. Solubility of these constituents may depend on the fineness of test sample also. Water cement ratio of concrete, extent of hydration and age of concrete, extended exposure to high *p*H of concrete, etc, may affect the solubility of some minerals.

3.1 Substances Affecting Calcium Oxide Procedure

The materials that are soluble even in dilute and cold hydrochloric acid like limestone, marble, dolomitic limestone, calcareous sandstone, chert, caliche encrusted and calcite dolomite coated rocks may lead to bias in results.

Following may be soluble depending on age and *p*H of concrete; weathered or altered plagioclase feldspar, caliche encrusted rocks, altered volcanic rocks (with calcareous inclusion) and many other calcium containing rocks.

Every percent of soluble calcium oxide that is contributed by soluble aggregate or mineral admixture will bias results of cement content higher by approximately 1.6 percent.

The estimated cement content by this method may be in error up to 5 percent if blank cement samples are available. In the absence of cement samples, total variation may go up to 10 percent because of presence of performance improvers in the cement.

3.2 Substance Affecting the Soluble Silica Procedure

Following may be soluble depending on age and *p*H of the concrete whether the aggregate is glassy or crystalline, weathered or strained; and the fineness of mineral: chert, opal, chalcedony, glassy volcanic rock, strained quartz, quartzite, catcalastic rocks (mylonite, phyllonite) gneiss, schist and many other soluble silicon containing rocks or minerals.

Every percent of soluble silica contributed by aggregate or mineral admixture will result in an increase in the measured Portland cement content by approximately 4.7 percent.

If part of silica fume is soluble under experimental conditions, the calculated cement content values shall be slightly biased on the higher side.

The estimated cement content by this method may be in error up to 10 percent if blank cement samples are available. In the absence of cement samples, total variation may go up to 15 percent because of presence of performance improvers in the cement.

4 REAGENTS

a) *Hydrochloric acid* — Approximately 3.3 N. Add 200 ml hydrochloric acid (specific gravity 1.19) to 600 ml distilled water.

b) *Sodium hydroxide*— 1 N. Dissolve 20 g of NaOH in 500 ml distilled water.

c) *Sulphuric acid* — Specific gravity 1.84.

d) *Hydrofluoric acid* — 40 percent.

5 PREPARATION OF SAMPLE

Every precaution shall be taken to have the sample of concrete used for analysis truly representative of material under consideration. A single concrete core taken through the entire depth of concrete, having a minimum length and diameter of four times the nominal maximum size of aggregate shall be appropriate. The recommended mass of concrete for this test is 5 kg. This mass should be obtained from more than one core when concrete is not thick enough and a single core weighs less than 5 kg. As an alternate, lumps of concrete weighing a minimum of 5 kg and meeting dimensional requirement as maintained for cores, shall be acceptable for the test. In this case at least two such lumps shall be provided for testing.

Following information about the materials shall be provided, while submitting the samples for analysis (if the samples are drawn from an ongoing/recently completed work):

a) Type of cement used;

b) Nature and extent of performance improver used;

c) Nominal maximum size of aggregate used;

d) Mineral composition of the aggregate, if available (the details may be confirmed by petrography); and

e) Mineral admixtures added in the concrete, if any, with details, if available.

If the samples are drawn from such a location that no information is available, it shall be mentioned. If the cement sample of cement used is available, the same shall be provided for better assessment. If cement sample is not provided, cement content determined by this method will be the content of ordinary Portland cement without performance improvers.

Crush the sample to pass 4.75 mm IS sieve, mix thoroughly and obtain a representative portion weighing about 1 kg by repeated riffle splitting. This portion shall be further crushed in jaw crusher or ground in disk pulverizer or rotary mill device so that all of the material passes 300 µm sieve. Care shall be taken to minimize the production of very fine particles. For this, use several passes through the sieve before regrinding the remainder of sample. After thoroughly mixing by coning about 10 times from one portion to another, a portion approximately 100 g shall be taken by quartering. Care shall be taken that finer fractions, which are richer in cement, are not discarded or lost during this process. This sample shall be carefully freed, by means of a strong magnet from particles of metallic iron abraded from jaw crusher or pulverizer.

Dry the crushed and ground material in an oven at 105 ± 5°C for 24 h and retain the sample in sealed container.

6 PROCEDURE FOR CEMENT CONTENT

6.1 It is advised that petrographic examination of concrete is conducted beforehand to assess the possible interferences and ensure logical interpretation of results. The study shall be specifically focused on detection of interfering materials as discussed above. Nature of cement used for concrete making that is, ordinary Portland cement, or Portland cement with performance improver shall also be established.

When petrographic examination indicates presence of siliceous aggregate or mineral admixture likely to be soluble in cold hydrochloric acid, soluble silica procedure should be avoided. In cases when aggregate is found to be containing significant amount of calcareous components calcium oxide procedure should not be used.

In all other cases, and also when petrographic examination is not conducted, both of the procedures shall be performed.

All analysis shall be done in triplicate and the average of the three values shall be used in calculating cement content.

6.2 Soluble Silica Procedure

Weigh accurately into each of three 250 ml beakers not less than 2.0 g portion of prepared sample. The quartered 100 g sample may be thoroughly mixed for preparing the representative 2 g sample. Moist it with a stream of ice cold distilled water. Add 100 ml of cold 3.3 N hydrochloric acid slowly with constant shaking with glass rod and maintain the temperature 3 to 5°C. Stir the content continuously or at least several times, while maintaining temperature (3 to 5°C) for 5 to 10 min or more to ensure proper decomposition using an ice bath or electric cooling apparatus.

NOTE — Observation of solution during dissolution may provide useful information. Considerable effervescence indicate a substantial amount of calcite or carbonated paste, delayed effervescence suggest a dolomitic aggregate. Lack of effervescence suggests the applicability of calcium oxide procedure.

Decant through the Buchner type porcelain funnel fitted snugly with two disks of quantitative filter paper for fine precipitate (Whatman No. 40) or weighed with crucible which contains a mat of short asbestos shreds practically insoluble in hydrochloric acid, in a filter flask.

Once the filtration has begun, care shall be taken that mat and accumulated residues do not dry out completely until the filtration process is complete. Regulate the suction so as to maintain a rapid rate of dropping during greater part of filtration. Retain as much of residue in the beaker as possible. Wash twice by decantation with hot distilled water, save the filtrate (A).

Transfer the residue collected on Gooch crucible or the filter paper from funnel to the beaker containing the balance of residue. Add 75 ml of 0.25 N sodium hydroxide to the residue while stirring and heat to about 75°C. Decant as before and wash with hot distilled water until the filtrate (B) is neutral to litmus. Combine the filtrates A and B.

Filtrate now contains silica in the form of silicic acid in true solution or in suspension in hydrochloric acid medium. Allow the filtrate to stand overnight to settle the suspended fine material; filter by decantation through a finer filter paper (Whatman No. 42). Add 10 ml of hydrochloric acid (sp. gr. 1.19) to the solution for neutralizing unreacted NaOH. Transfer the content to a suitable beaker with several rinsing of the filter flask. Evaporate to dryness with great care to minimize spattering. Bake at 110 ± 5°C for 1 h. Moisten with hydrochloric acid (sp. gr. 1.19) evaporate and bake again, take up for filtration in 75 ml of HCl (1:3). Heat to boiling, filter through an ashless filter paper (Whatman No. 40) and wash the residue with 50 ml hot dilute hydrochloric acid (1:99) and then with hot distilled water until washings are free from chloride. Determine the silica present in the sample in accordance with the procedure given in test method in IS 4032.

6.2.1 *Calculation*

Calculate the cement percentage in sample by dividing the percent silica (SiO_2) in the concrete by the percent silica (SiO_2) in the cement, and multiplying by 100. If the cement silica value is unknown, assume 21.4 percent for ordinary Portland cement (without performance improvers).

6.3 Calcium Oxide Procedure

Calcium oxide shall be determined by following procedure. Omit the determination, if it is known that aggregate contains substantial amount of calcareous component.

Using the filtrate from the removal of silica (*see* **6.2**), separate the ammonium hydroxide group and then determine calcium oxide in accordance with the test methods explained in IS 4032.

6.3.1 *Calculation*

Calculate the cement percentage in sample by dividing the percentage of calcium oxide in concrete by percentage of calcium oxide in cement and multiplying by 100. If the calcium oxide in cement is unknown then assume 63.5 percent for ordinary Portland cement (without performance improvers).

6.4 Cement content determined in terms of mass percentage can be converted to kg/m^3 of concrete (if required to be determined) by determining the dry density of a separate concrete sample having definite dimensions like a core sample of minimum diameter 4 times the aggregate and minimum height 4 times the maximum aggregate size and weighing minimum 0.5 kg .

7 REPORT

7.1 Report shall contain the user provided information as mentioned in **5**.

7.2 Results of petrography shall be reported with interpretation and opinion about possible interferences.

7.3 The cement content in the sample, mentioning the procedure used, shall be reported. If both procedures (soluble silica and calcium oxide) have been used, the lower of the two shall be reported and the procedure name shall be mentioned. All analysis shall be done in triplicate, and the average of the three values reported.

7.4 The average weight percentage of ordinary Portland cement in concrete shall also be reported, if relevant.

ANNEX A

(*Foreword*)

COMMITTEE COMPOSITION

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Panel for Revision of Indian Standards on Test Methods for Concrete, CED 2 : 2/P7

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(*Continued from second cover*)

b) two methods of analysis, that is, through determination of soluble silica and through determination of calcium oxide have been included, which may be appropriately chosen from or both may be performed, based on petrographic examination as detailed in the standard.

The composition of the Committee responsible for the formulation of this standard is given in Annex A.

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

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Eastern	: 1/14 C.I.T. Scheme VII M, V.I.P. Road, Kankurgachi KOLKATA 700054	2337 8499, 2337 8561 2337 8626, 2337 9120
Northern	: Plot No. 4-A, Sector 27-B, Madhya Marg CHANDIGARH 160019	265 0206 265 0290
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